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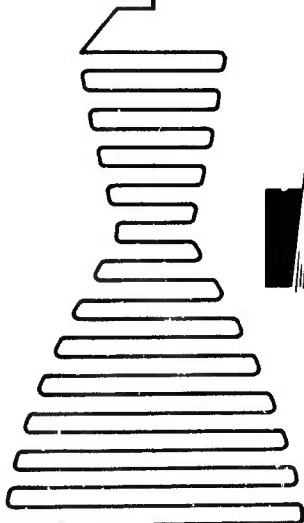
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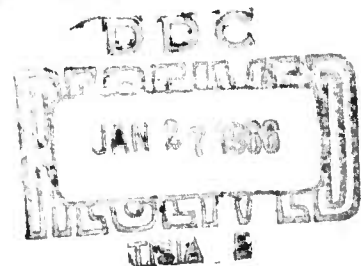
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R-5883-2

(Unclassified Title)

QUARTERLY PROGRESS REPORT,
INORGANIC HALOGEN OXIDIZERS
(1 September through 30 November 1964)

Group 4
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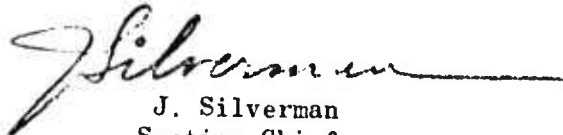
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FOREWORD

The research reported herein was supported by the Advanced Research Projects Agency and was monitored by the Office of Naval Research, Power Branch, Code 429, Washington 25, D.C., with Mr. Richard Hanson as Scientific Officer. This report was prepared under G.O. 8614 in compliance with Section H of Contract Nonr 4428(00) under ARPA Order No. 23, and covers the period 1 September through 30 November 1964.

The Responsible Engineer for this program is Dr. D. Pilipovich. Dr. H. F. Bauer and Dr. C. J. Schack are full-time associates. The work was carried out in the Chemistry Section of the Rocketdyne Research Department.

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ABSTRACT

The reaction of solid FClO_2 with excited fluorine at -196°C gave rise to ClF_5 . The absence of ClF_3 in the reaction products led to the premise that metastable species involving ClOF moieties may have been intermediates in the formation of ClF_5 .

Solid N_2O_4 at -196°C reacted with excited fluorine as NO^+NO_3^- . The principal products from the reaction were NF_3O and NO_3F .

A complete examination of the $\text{ClF}_5\text{-FNO}_2$ and $\text{ClF}_5\text{-FNO}$ systems by nuclear magnetic resonance (n.m.r.) spectroscopy showed conclusively that there were no interactions at -108 to -60°C . Chemical shifts and splitting constants in the mixture were virtually identical to those of the pure components.

The reaction of $\text{ClF}_4^+\text{AsF}_6^-$ with H_2O , N_2O_3 , and $(\text{EF}_3\text{CO})_2$ failed to give ClF_3O or $\text{ClF}_2\text{O}^+\text{AsF}_6^-$.

(Confidential Abstract)

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DISCUSSION

DISCHARGE REACTIONS

Attempts to prepare new F-Cl-O compounds by discharge reactions were continued. The principal effort used FClO_2 as the substrate. The techniques utilized were essentially those evolved earlier in this program, and were described in the first quarterly report (Ref. 1). It was shown previously that the frozen reactant, if much removed from the discharge zone, is not subjected to a high concentration of excited fluorine. Therefore, in these experiments the substrate was condensed immediately adjacent to the discharge zone or even partially in it.

When FClO_2 was fluorinated at liquid nitrogen temperature in this manner, it was observed that the original white to yellow solid developed a purple ring in that portion of the material nearest the discharge. At the conclusion of a run the cooling bath was removed and the volatile materials were immediately recondensed into the vacuum line at -196°C . Upon warming to room temperature to measure the product gas volume and the recooling, some gases not condensable at liquid nitrogen temperature always remained. Presumably this noncondensable gas is fluorine and/or oxygen. After pumping this gas off, the remaining product gases were allowed to vaporize, and an infrared spectrum was taken.

Along with a trace of SiF_4 produced by attack on the glass, the products were unreacted FClO_2 , contaminant ClO_2 , and an appreciable concentration of ClF_5 . It appears then that the discharge fluorination has resulted in the conversion of FClO_2 to ClF_5 . Such a displacement by fluorine of oxygen bonded to chlorine has not been reported previously. Any reasonable interpretation of this result cannot be based upon a direct displacement reaction. Much more likely is the formation of an intermediate

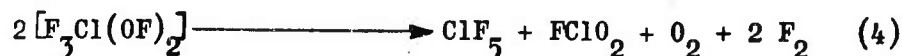
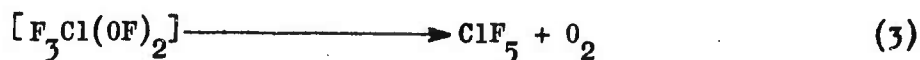
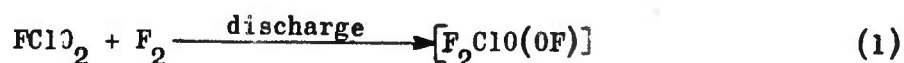
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unstable hypofluorite compound. Such structures, while stable at low temperature, might easily rearrange upon warming to give both the observed ClF_5 and noncondensable gases.

Although purely hypothetical, the following reaction scheme indicates some of the possible intermediate hypofluorites and shows how they might give rise to ClF_5 :



Since no ClF_3 was found, it is probably not involved in the conversion of FClO_2 to ClF_5 . The absence of ClF_3 also precludes Cl_2 as an intermediate. In discharge reactions of F_2 and Cl_2 , the ratio of ClF_3 to ClF_5 found was always on the order of 10 to 1.

To determine the generality of displacing oxygen from XO_2 species by discharge fluorination, this reaction was carried out with sulfur dioxide. If any hypofluorites of sulfur are formed, they should be stable to warming (e.g., SF_5OF), and thus the intermediate character of Cl-OF species in the overall reaction could be demonstrated. Under the same conditions employed with FClO_2 , it was determined that SO_2F_2 and SF_6 were produced. Thus, fluorination and displacement did occur, but intermediate OF species were not isolated.

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In earlier experiments with Cl_2O and air-contaminated fluorine, it was noted that this discharge reaction also gave rise to some noncondensable gases upon warming to room temperature. Since in the absence of air no such gases were observed, it is possible that an unstable F-N-O compound such as $\text{F}_2\text{NO}(\text{OF})$ might be the source. To test this possibility several discharge runs were conducted using solid N_2O_4 as the reactant. The results of these reactions were somewhat ambiguous. In addition to a large amount of unreacted N_2O_4 , some FNO_3 always was present with little or no FNO_2 found. This suggested that the N_2O_4 had reacted as though it were present in the ionic form $(\text{NO}^+\text{NO}_3^-)$. However, the fact that NF_3 was at times formed instead of the expected NF_3O is perplexing. If the reaction proceeds through NO^+NO_3^- predominantly, as appears to be the case, then it is not clear why in some instances NF_3O is formed and in others only NF_3 is formed. The predominant reaction path, however, is:



Although present to a lesser degree than in the case of FClO_2 , it was again found that the discharge reaction products upon first warming in the vacuum line gave some gases not condensable by liquid nitrogen.

The reaction of ClF_5 in a discharge at -80°C was examined. It was hoped that ClF_2 or ClF_4 radicals might be generated. Such radicals might react with the glass reactor to form new F-Cl-O compounds or couple to yield compounds such as $(\text{F}_4\text{Cl})_2$. The coupling reaction is analogous to the preparation of B_2Cl_4 in a mercury electrode discharge apparatus (Ref. 2).

In a series of experiments beginning at the lowest voltage at which a discharge could be maintained and then at higher voltages, it was determined that no new species were obtained. At the lower voltages, the

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ClF_5 was essentially unchanged. At higher voltages, the ClF_5 was completely destroyed, giving mainly F_2 and Cl_2 . The other products were ClO_2 , FClO_2 , and SiF_4 .

The discharge reactions reported have indicated several areas in which new metastable compounds may have been produced. The formation of non-condensable gases during product workup may be indicative of the presence of O_2F_2 . However, the characteristic color of O_2F_2 was never observed in any of the reaction mixtures. These discharge studies will be resumed when appropriate low-temperature infrared equipment is available for a more complete examination of the reaction products.

METATHETICAL REACTIONS

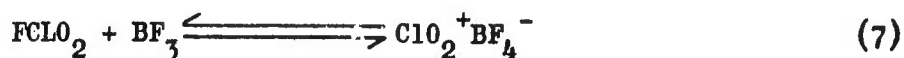
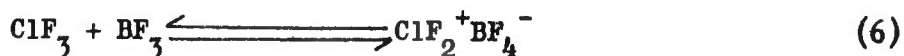
This first of the proposed metathetical reactions to be studied is that involving ClF_3 and HNF_2 . This reaction had been studied at Rocketdyne in the past, but the presence of the impurities FClO_2 and ClO_2 in the ClF_3 has obscured its course. It had been postulated that the NF_3O formed in these early experiments and also the deflagrations often encountered were due to the reaction of these impurities (Ref. 3). Therefore, to ascertain the true course of the ClF_3 - HNF_2 reaction, the preparation of pure ClF_3 was undertaken.

Because simple physical methods are inadequate, it was necessary to form a complex with ClF_3 from which the impurities could be removed. Subsequent removal of the complexing agent would then result in pure ClF_3 . Three complexes were studied. The complex with BF_3 forms readily at -80°C , at which temperature the ClO_2 is readily pumped off the solid complex. However, the FClO_2 contaminant also complexes with BF_3 and thus is not separated.

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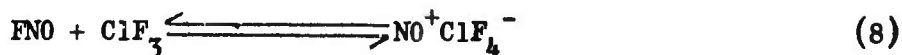


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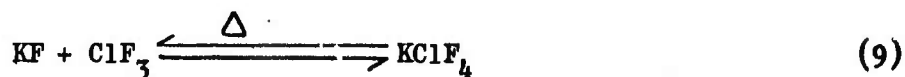
It was rather surprising that FClO_2 could not be removed from the mixture since the complex, $\text{ClO}_2^+ \text{BF}_4^-$, has been reported to have some volatility at -80°C (Ref. 4).

The complex with FNO was also formed at -80°C , and because of the lower volatility of the solid, the impurities could be more readily stripped away at -46°C :



However, it was then discovered that even though the solid is completely dissociated into its component gases at ambient temperature, these gases regenerate the solid complex despite the most rapid cooling. Therefore, subsequent fractionation fails to secure a separation.

It was finally determined that pure ClF_3 could be derived from the KF complex:



It was formed from solid KF and liquid ClF_3 at room temperature in a stainless-steel cylinder. This technique is analogous to that recently reported by Whitney et al. (Ref. 5). Since the KClF_4 is nonvolatile at room temperature, the excess ClF_3 and impurities are pumped out of the cylinder readily. By heating the bomb and its solid contents several minutes under vacuum, high-purity ClF_3 is obtained.

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When first utilized, it was thought that this method was only upgrading the impure material since some small amounts of ClO_2 and FCIO_2 were still observed in the infrared spectrum of the ClF_3 . This difficulty was soon clarified when it was determined that the impurities were arising during the charging of the infrared cell with the ClF_3 . Decomposition was occurring by reaction of the ClF_3 with the inside of the metal ball joints connecting the cell to the vacuum line. This joint must necessarily be exposed to the air when running a spectrum and consequently becomes de-passivated each time the cell is used. When passivated prior to charging with ClF_3 , no further decomposition was noted, and a true spectrum of the ClF_3 was obtained.

Initial experiments using equimolar amounts of ClF_3 and HNF_2 or excess ClF_3 have been conducted without deflagration. When warmed to room temperature and then re-cooled and fractionated, it was found that all the HNF_2 was consumed, giving rise to ClNF_2 , N_2F_4 , and NF_3 . These are the same products obtained previously (Ref. 3). Most of the ClF_3 remained unchanged. When mixing and while at low temperature, the reactants were kept in a Teflon trap. Over a short period at -80°C there was no detectable increase in vapor pressure; however, warming to ambient temperature with subsequent cooling to -80°C gave higher than predicted vapor pressure. Since the vapor was exposed to metal, it may be that one reaction of the HNF_2 was its decomposition on the metal surface. To circumvent this possibility, future reactions of these materials will be conducted in a nonmetal system.

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 ClF_5 -OXYNITROGEN FLUORIDES

Novel routes to new N-O-F, Cl-O-F, or Cl-F compounds may be realized by the fluorination of the hypothetical anions F_2NO^- , F_2NO_2^- , ClF_4O^- , or ClF_6^- .

Interactions between ClF_5 and either FNO or FNO_2 at low temperatures were considered as possible sources of the desired species:



Even slight interactions between these moieties should be detectable by low-temperature n.m.r. F^{19} spectra of the mixtures. The results of the n.m.r. study are shown in Table 1. All n.m.r. spectra were taken in quartz n.m.r. tubes flamed out under high vacuum before loading.

The chemical shifts and splitting constants of the mixture components were essentially the same as those of the pure component. The recurrent resonance near 100 ppm was attributed to FClO_2 rather than F_3ClO since some splitting would be expected from the nonequivalent fluorines predicted for F_3ClO . The appearance of FNO_2 in the $\text{FNO}-\text{ClF}_5$ mixture was noted, along with considerable N_2O and N_2O_3 . The presence of N_2O caused troublesome bubbling in the tube and necessitated operation at -108°C rather than -80°C . No evidence for interactions leading to stable anions or new species was found. The absence of any interaction between either FNO_2 or FNO with ClF_5 by n.m.r. analysis is in sharp contrast to the reports of other workers. The reaction of ClF_5 and FNO_2 has been reported to yield a complex at -80°C which is capable of a reversible dissociation (Ref. 6). With FNO, ClF_5 has been reported to form a solid complex that is dissociated at -20°C (Ref. 7). Attempts at checking interactions by vapor pressure measurements were unsuccessful due to partial decomposition of

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TABLE 1

NUCLEAR MAGNETIC RESONANCE SPECTRA OF $\text{FNO}-\text{ClF}_5$ AND
 $\text{FNO}_2-\text{ClF}_5$ MIXTURES

Components	Chemical Shift Relative to F_2 , ppm	J Value, cps	Assignment	Temperature, C
FNO_2	36	118		-41
	34	116		-59
FNO	-60	broad unresolved peak		-80
ClF_5	164	142	doublet	-80
	1	142	quintet	-80
$\text{FNO}_2-\text{ClF}_5(1:5)$	167.5	133	ClF_5 doublet	90
	-3.5	130	ClF_5 quintet	
	30	123	FNO_2	-60
	95	singlet	FCIO_2	
	167.5	130	ClF_5 doublet	
	-2.5	130	ClF_5 quintet	
	31.5	118	FNO_2	
	95	singlet	FCIO_2	
	167	145	ClF_5 doublet	-108
	0	145	ClF_5 quintet	
	31	130	FNO_2	
	101	singlet	FCIO_2	
$\text{FNO}-\text{ClF}_5(1:2)$	-62	broad unresolved peak	FNO	

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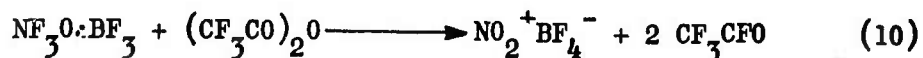
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the mixtures in metal. When FNO and ClF_5 were allowed to combine at ambient temperature in metal, partial reaction gave N_2O , FNO_2 , Cl_2 , and NF_3O as products.

REACTIONS OF ClF_5 COMPLEXES

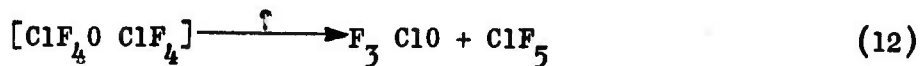
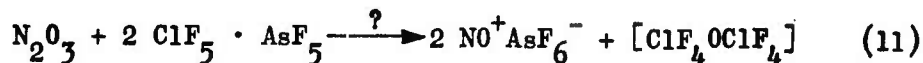
Several attempts have been made to react the $\text{ClF}_5\text{-AsF}_5$ complex with oxygen-containing materials in the hope of utilizing the increased electrophilicity of the chlorine atom and thus producing new Cl-O-F compounds. The reaction of ice with the $\text{ClF}_5\text{-AsF}_5$ complex produced ClF_5 and AsF_3O when the mixture was warmed slowly from -80°C . It is possible that the initial formation of HF converts H_2O to H_3O^+ , which makes oxygen attack by ClF_4^+ unlikely.

The reaction of $\text{ClF}_5\text{-AsF}_5$ with trifluoroacetic anhydride was attempted in the hope of obtaining an extension of the known reaction:



Although ClF_5 reacted smoothly with $(\text{CF}_3\text{CO})_2\text{O}$ at 20°C to produce FClO_2 and CF_3CFO , the $\text{ClF}_5\text{-AsF}_5$ complex reacted with sufficient vigor below 0°C to pop open the all Kel-F/Teflon reactor so that no products were recovered.

The low-melting compound N_2O_3 was combined with $\text{ClF}_5\text{-AsF}_5$ at -80°C in the hope of producing the following reactions:



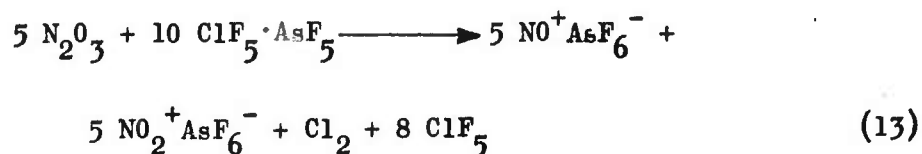
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The volatile products found were Cl_2 and ClF_5 , but no FClO_2 or ClO_2 . Pumping on the solid residue, with warming, liberated some FNO_2 . Analysis of the residue showed the absence of chlorine. Fluorination of the N_2O_3 appears to have occurred according to the following equation:



The reactions of ClF_5 with Ag_2O or HgO have consistently produced good yields of FClO_2 . Since in the synthesis of Cl_2O from Cl_2 , yellow HgO is successful while red HgO is not, it was felt that the reaction of ClF_5 with yellow HgO under mild conditions might produce the intermediate product F_3ClO rather than FClO_2 . A slow stream of ClF_5 diluted with N_2 was passed through a Kel-F U-tube packed with a powdered mixture of yellow HgO and Kel-F at 0 C.

Reaction took place with the Kel-F, giving Cl_2 , CF_4 , CF_3Cl , and $\text{CF}_3\text{CFCF}_2\text{Cl}$, as well as other unidentified halocarbons. No Cl-O-F products were detected. The substitution of copper for Kel-F powder as an HgO diluent is planned.

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EXPERIMENTAL DETAILS

Chloryl fluoride was prepared from silver oxide and ClF_5 (Ref. 1). Nitrogen tetroxide, sulfur dioxide, and boron trifluoride were obtained from the Matheson Company. Nitrosyl fluoride was prepared fresh from the reaction of 1 mole fluorine with 2 moles of nitric oxide at -196°C . Difluoramine was prepared by acidification of an aqueous solution of N,N-difluorourea and fractionated before being used.

All discharge experiments were carried out using the previously mentioned apparatus (Ref. 1) with a 6-inch electrode gap. All runs had an approximate duration of 1 hour except those with ClF_5 . In these runs the time was varied from a few minutes to 2 hours. In all cases, the fluorine pressure was maintained at 50 to 60 millimeters throughout the experiment.

The preparation of KClF_4 from ClF_3 and KF was not quantitative. Depending partly upon the length of mixing and also the amount of excess ClF_3 , conversions of 50 to 80 percent were achieved. In addition to the long reaction room temperature, satisfactory yields of KClF_4 could be obtained by heating the reactants for 2 hours at 50 to 60 $^\circ\text{C}$. It was essential that the reaction cylinder be shaken during the reaction to achieve good yields.

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